



PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.: 09/918,158
Filing Date: July 30, 2001
Applicant: Robert A. Dichiarà Jr.
Group Art Unit: 1731
Examiner: Christopher A. Fiorilla
Title: OXIDE BASED CERAMIC MATRIX COMPOSITES
Attorney Docket: 7784-000146

Director of the United States Patent and Trademark Office
P.O. Box 1450
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DECLARATION UNDER 37 C.F.R. § 1.131

Sir:

I hereby declare under penalty of perjury as follows:

1. That I am the sole inventor of the above-identified application.
2. That the invention was conceived and at least partially reduced to practice in this country prior to February 24, 1994, the filing date of the United States Patent No. 5,422,331 to Galligan et al. and prior to December 20, 1996, the filing date of the United States Patent No. 5,958,583 to Rorabaugh et al. In addition, this invention was

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conceived and at least partially reduced to practice in this country prior to December 15, 1999, the filing date of United States Patent No. 6,497,776 to Butler et al., and prior to March 3, 1998, the filing date of United States Patent No. 6,110,439 to Deshpande et al.

3. I am the author of the notebook whose cover page is attached at Exhibit A. Pages from this notebook are attached as Exhibits B and C and the information contained within this notebook was either prepared by myself or under my direction.

4. That the invention was conceived and/or reduced to practice prior to February 25, 1994, as evidenced by the notebook page attached as Exhibit B. Exhibit B illustrates at least the initial conception and reduction to practice of a composition embodied by at least claim 1. A second page from the notebook is attached as Exhibit C and shows reduction to practice of a further embodiment of the invention claimed in at least claim 1 prior to February 25, 1994.

5. That the invention has never been abandoned, suppressed, or concealed.

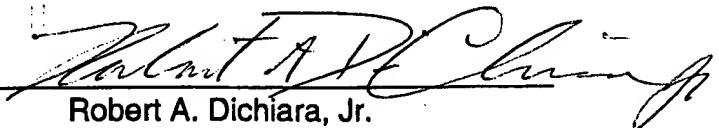
6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are being made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false

statements may jeopardize the validity of the application, and patent issuing thereon, or any patent to which this verified statement is directed.

Dated: _____

8/25/04

Robert A. Diciara, Jr.



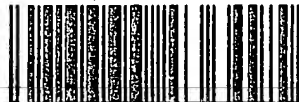
Department REC-100
Subject CME (M-106)
Name R. J. O'Hara
Address _____



43-649

**Laboratory
Research Notebook**

Dennison National Company, Holyoke, MA 01041



11" x 9 1/4" 4 x 4 Quad.
200 Sheets - 100 Sets

0-73333-43649-5

Exhibit A

Charge #: IR-53704

VARIABLES		
A	Mullite (Baikalox Submicron A) $2\text{O}_3/\text{SiO}_2: 1.75$	Silica Sol/Mul 1.3

LAMINATE SIZE: 6.5" (Warp direction) x 9.5" (Fill direction) untrimmed

Plies: 8

- a) Heat clean a piece of 8HS Nicalon fabric 39" wide (Fill direction) by 13" long (Warp direction).
 b) Make up mix and use just after mixing or remix before use. Put 500 grams of Silica Sol (Nalco 2327) into the small ball mill and add 375 grams of submicron Baikalo Mullite ($\text{Al}_2\text{O}_3/\text{SiO}_2: 1.75$) mix for 0.5 hours. Add 1.5 grams of Dow Corning antifoam 1410. Mix for 3.5 hours use mixture after this point. If observe foaming when opening up mill or viscosity not right for prepreging let Bob D. know. If cannot use that day, ball mill mixture again for 2 hours before use.
 c) Hand prepreg the fabric try and achieve about 33 % fiber to matrix ratio and lay-up 8 wet plies nested together, 6.5" (Warp direction) x 9.5".

d) Fabric Wt. 127.7 g.Prepreg Wt. (actual) 307.1 g.Fiber/Matrix ratio = Fabric Wt./Prepreg Wt. (actual) x 100 = 41.6 %.

e) Press cure the laminate

Apply 200 psi immediately and heat press to 200°F hold for 1/2 hour.

Heat press to 220°F at 1°F/min and hold for 1/2 hour.

Heat press to 350°F at 1°F/min. and hold for 1/2 hour.

Remove laminate for post curing. 2000°Ff) Post cure: To 1500°F 2 hr at 5 to 10°F/minute.

g) Do physicals: % Porosity, % Matrix and % Fiber.

h) Cut laminate into flexure samples (0.5" x 5.5" [Warp direction]).

i) Heat treat samples placing samples into furnace at temperature and remove to room temperature.

m) Test Samples:	Heat Treated	Testing Temperature
4 Flexure	None	RT
4 Flexure	1500°F/1hr	1500°F
4 Flexure	2000°F/1hr	2000°F
4 Flexure	2000°F/1hr	RT
	<u>2400</u>	

COMMENTS:

Program - hold was planned set out 180 min. 7:50pm went to 210°F for 1/2 hr.

Then to 425°F / 1°F/min hold 1 hour. on 10-7-93 7:30AM removed from
 245° press. Panel looked excellent, uniform surface and .105" thick
 seems hard. Post cured to 2000°F for 2 hrs. Part hardens up
 within 5 minutes at 180°F. Mixture putties-up after one
 day in a closed container

There is a key
 here for other wo
 as well. Good den
 laminate. # submic
 material
 # 2 viscosity slurry
 # 3 bleeder package

May want to repeat with H_2CO_3

May want to try sub
 mullite in phosphate
 chemistry as well. See if
 submicron is major effect
 on viscosity

Charge #: IR-569030

VARIABLES

A	Alumina (SM-8)	B. Silica Sol/Alumina	1.3
C.	Nextel 610 (alumina fiber)		

LAMINATE SIZE: 6.5" (Warp direction) x 9.5" (Fill direction) untrimmed
Plies: 8

a) Use heat cleaned Nextel 610 (8HS fabric) 39" wide (Fill direction) by 13" long (Warp direction).
b) Make up mixture and use just after mixing or remix just before use. Put 500 grams of Silica Sol (Nalco 2327) into the small ball mill and add 375 grams of submicron Baikalo SM-8 (Al₂O₃) mix for 0.5 hour. Add 1.5 grams of Dow Corning antifoam 1410. Mix for 3.5 hours use mixture after this point. If observe foaming when opening up mill add 0.5 grams of antifoam 1410, tumble for 5 minutes and record on sheet. If problems or viscosity not right for prepregging let Bob D. know. If cannot use that day, ball mill mixture again for 2 hours before use.

If slurry still appears good from Alum/Sol-I-1 you can use slurry again if you high shear at 5000 rpm's for 5 minutes (please make note on sheet of the material you used).

c) Hand prepreg the fabric try and achieve about 38-40 % fiber to matrix ratio and lay-up 8 wet plies nested together, 6.5" (Warp direction) x 9.5".

d) Fabric Wt. 108.8 g,

Prepreg Wt. (actual) 244.7 g.

Fiber/Matrix ratio = Fabric Wt./Prepreg Wt. (actual) x 100 = 44.2 %.

e) Make up cork dam set up with bleaderlease C on both sides like used in Mul-IV-1.

f) Press cure the laminate,
use one layer of armolon and one layer of pink release glass on both sides
Apply 200 psi immediately and heat press at 2°F/min. to 180°F hold for 1.5 hour.
Heat press to 210°F at 1°F/min and hold for 1 hour.
Heat press to 425°F at 5°F/min. and hold for 1 hour.
Remove laminate for post curing.

g) Post cure: To 2000°F/2 hr at 5 to 10°F/minute.

h) Reinfiltarate with SiO₂ Sol. (Nalco 2327) 2 times.

i) Weigh panel to start and reinfiltate panel with Silica Sol under vacuum (30" Hg) for 30 min.. Remove panel from sol and place in oven at 300°F for 30 min.. Pull panel out of oven and when at room temperature wipe off excessive silica powder off of surface and weigh. Repeat process 2 times. Weight as made 136.5 g, 1st infiltration 141.2 g, 2nd infiltration 143.7 g.

j) Fire panel to 2000°F for 2 hours. After firing _____ g.

k) Cut panel in half and dry at 220°F.

l) Reinfiltarate half the panel with SiO₂ Sol. (Nalco 2327) for 2 more times.

m) Weigh panel to start and reinfiltate panel with Silica Sol..

Weight as made _____ g, 3rd infiltration _____ g, 4th infiltration _____ g.

n) Fire panel to 2000°F for 2 hours. After firing _____ g.

o) Do physicals: % Porosity, % Matrix and % Fiber.

p) Cut laminate into flexure samples (0.5" x 5.5" [Warp direction]).

q) Heat treat samples placing samples into furnace at temperature and remove to room temperature.

r) Mechanical test both 2 and 4 times infiltrated panels.

Test Samples:	Heat Treated	Testing Temperature
4 Flexure	None	RT
4 Flexure	1800°F/1hr	1800°F

Exhibit C

COMMENTS:

yields 800 gms of slurry after ball milling
or 480 ml

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